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(21) International Application Number: PCT/GB00/01475 (22) International Filing Date: 17 April 2000 (17.04.00) (30) Priority Data: 9909444.3 23 April 1999 (23.04.99) GB 9927803.8 24 November 1999 (24.11.99) GB (71) Applicant (for all designated States except US): IMPE- RIAL COLLEGE OF SCIENCE, TECHNOLOGY AND MEDICINE [GB/GB]; Exhibition Road, London SW7 2AZ (GB). (72) Inventors; and (75) Inventors/Applicants (for US only): DURRANT, James, Robert [GB/GB]; 44 Standen Road, London SW18 5TQ (GB). HAQUE, Saif, Ahmed [GB/GB]; 38 Lichfield Road, Northwood Hills, Middlesex HA6 1LZ (GB). MARTIN, Rainer, Eugen [AT/GB]; 23 Midwinter Place, Hamilton Road, Cambridge CB3 9JH (GB). HOLMES, Andrew, Bruce [AU/GB]; 19 Newton Road, Cambridge CB2 2AL (GB). (74) Agent: ROBINSON, Nigel, Alexander, Julian; D Young & Co., 21 New Fetter Lane, London EC4A 1DA (GB).		(81) Designated States: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG). Published Without international search report and to be republished upon receipt of that report.	
(54) Title: PHOTOVOLTAIC DEVICES			
(57) Abstract			
<p>A photovoltaic device (2) is described comprising a porous film of titanium oxide nanocrystalline particles (4) intercalated with an ionic conducting material (polymer) electrode (8). The ionic conducting polymer electrode (8) may be formed of a mixture of polyethyleneoxide, lithium iodide and iodine. The nanocrystalline titanium oxide particles (4) are dye sensitised. In operation a photon is absorbed injecting an electron into the conduction band of the titanium oxide (4). A hole is passed to the ionic conducting polymer (8). The electron is conducted through the titanium oxide nanocrystalline particles (4) and the hole is conducted through the ionic conducting polymer electrode (8). The cell may be unsealed without rapid evaporation of iodine.</p>			

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5

PHOTOVOLTAIC DEVICES

This invention relates to the field of photovoltaic devices.

Efficient dye sensitised photovoltaic devices employing nanocrystalline metal oxide
10 films were first reported in 1991, see B. O'Regan and M. Gratzel, Nature, 1991, 353, 737.
Such cells can achieve solar to electrical energy conversion efficiencies of up to 10%.
There is currently extensive commercial R&D aiming to develop photovoltaic devices
based upon this design, centred around Prof. Gratzel's group in EPFL, Switzerland.
Previous investigations have searched for the identity of the optimum sensitizer dye for this
15 technology.

An important limitation in the design of dye sensitised PV devices currently being
commercialised is the requirement for a electrically conducting liquid component (an
electrolyte). It has been proposed to replace this liquid with a solid state electrolyte
20 analogue. The important requirements for such solid state analogues are good device
energy conversion efficiency, good stability and low sealing requirements. Gratzel and co-
workers have reported that an organic material, OMeTAD developed for the Xerographic
industry, is one such material, although the efficiency of the solid state device produced
with this material was limited to < 0.8 %, see Back et al., Nature 1998, 395, 583. Other
25 proposals have considered conducting polymers, but have to date achieved efficiencies
even less than the above, see Murakosh, et al., Chem Letts., 1997, 471. Polymer gel
electrolytes have also been shown to achieve high efficiencies, however such system retain
a solvent phase and therefore still require sealing, and cannot therefore be regarded as truly
solid state, see Cao et al., J. Phys. Chem, 1995, 99, 17071. There has been a report of the
30 use of solid state ionic commercial rubber, but efficiencies achieved were very low (0.1%),
see Nogueira et al., Abstracts IPS- 12.

Various photovoltaic and battery cells are described in US-A-5.441.827, US-A-
5.438.556, US-A-4.520.086, WO-A-97/08719 and WO-A-93/20569.

5 Viewed from one aspect the present invention provides a photovoltaic device including an ionic conducting material as a solid state electrode.

 Ionic conducting materials have been found to provide good energy conversion efficiencies, good stability and require little or no sealing.

10

 Preferably the ionic conducting material comprises molecular, oligomer or polymeric components.

 The use of ionic conducting materials as an electrode in a photovoltaic device is particularly well suited to photovoltaic devices including a nanocrystalline particle film.

15

 The nanocrystalline particle film could have various forms, but it is preferably a nanocrystalline metal oxide film.

20

 Metal Oxide films, such as titanium oxide, are well suited to having electron injection into the titanium oxide conduction band followed by electron conduction through the titanium oxide particles to a electrode.

 In the context of a photovoltaic device including a nanocrystalline particle film, the ionic conducting material is preferably intercalated with the nanocrystalline particle film such that it can act as an electrode to carry away a hole following the absorption of a photon and the injection of an electron into the conduction band of the nanocrystalline particle.

25

 Conversion efficiencies of such devices are significantly improved when the nanocrystalline particle film is dye sensitised.

30

 The suitability of the ionic conducting material for use as an electrode is significantly enhanced when the ionic conducting material is doped.

5

The conductivity of the ionic material may be further enhanced by the addition of electronic hole transporting components, including, for example triaryl amine compounds. Such hole transporting components may or may not be covalently bound to other components of the ionic conducting material.

10

The effectiveness of the ionic conducting material as an electrode is further enhanced when it is mixed with redox active components, including salts, such as lithium iodide.

15

The ionic conducting material can have various forms. A particularly preferred ionic conducting material is polyethyleneoxide and variants thereof.

20

Surprisingly high efficiency, high stability and low sealing requirements have been found in photovoltaic devices including polyethyleneoxide, iodine and lithium iodide as a mix providing the role of an electrode.

25

A particularly suitable disposition for the various components of the photovoltaic devices is one in which the ionic conducting material and the nanocrystalline particle film are disposed on one of a conducting glass substrate and a conducting plastic substrate.

30

Incident photons can pass through the transparent plate where they are absorbed by the photovoltaic device leading to electron transport via the nanocrystalline particle film and hole transport via the ionic conducting material.

The present invention is particularly well suited for use in photovoltaic devices including iodine and that are unsealed. Iodine is normally considered relatively volatile and it is surprising that these devices show good stability obviating the need for sealing.

5 The ionic conducting polymer and nanocrystalline particle film layer can have various thickness'. Preferably the thickness of this layer is between five and fifteen micrometers and still more preferably the thickness of the layer is substantially ten micrometers.

10 The nanocrystalline particle film could be formed of various different substances providing that they have the correct physical and electrical properties. A particularly preferred option is the use of titanium oxide nanoparticles. Preferably such nanoparticles are between five and fifty nanometers in diameter, and still more preferably substantially ten nanometers in diameter. An alternative option is the use of zinc oxide nanoparticles.

15

Viewed from another aspect the present invention provides a method of making a photovoltaic device, said method comprising the steps of:

forming a porous nanocrystalline particle film; and

forming an ionic conducting material intercalated with said nanocrystalline particle
20 film, said ionic conducting polymer film serving as a solid state electrode.

It is surprising that the ionic conducting material with its relatively high molecular mass is able to intercalate effectively with a porous nanocrystalline particle field and so provide good function as an electrode within the photovoltaic device.

25

The porous nanocrystalline particle film could be formed in a number of different ways. Preferably, the nanocrystalline particle film is formed by one of:

use of an aqueous sol/gel;

thermolysis in a high boiling point solvent;

30 chemical vapour deposition; spray pyrolysis; and
spreading and spin coating.

An embodiment of the invention will now be described, by way of example only, with reference to the accompanying drawings in which:

5

Figure 1 schematically illustrates a photovoltaic device according to one example embodiment of the invention.

Figure 1 shows a photovoltaic device 2. The photovoltaic device 2 includes a
10 porous film of titanium oxide nanocrystalline particles 4. These titanium oxide
nanocrystalline particles 4 (or zinc oxide nanoparticles) may be formed by one or more of
the use of an aqueous sol/gel, thermolysis in a high boiling point solvent, chemical vapour
deposition, spray pyrolysis and spreading and spin coating. The titanium oxide
nanocrystalline particles preferably have a diameter between five and fifty nanometers and
15 still more preferably of substantially ten nanometers. The titanium oxide nanocrystalline
particles 4 are dye sensitised with a dye layer 6. This dye layer is preferably formed of one
or more of types of sensitiser dye, such as bipyridyl, porphyrin and phthalocyanine
sensitiser dyes.

20 An ionic conducting polymer (or material in molecular or oligomer form) electrode
8 is intercalated with the porous film of titanium oxide nanocrystalline particles. The ionic
conducting polymer 8 is preferably a mixture of polyethyleneoxide, lithium iodide and
iodine and variants thereof. The ionic conducting polymer 8 may be further adapted to its
role as an electrode for conducting holes by doping with triaryl amine components. The
25 porous nanocrystalline titanium oxide particle film 4 and the intercalated ionic conducting
polymer 8 preferably form a layer of between five and fifteen micrometers in thickness and
still more preferably of substantially ten micrometers in thickness. One face of this layer is
bounded by a glass plate 10. A platinised or tin oxide film 12 on the glass oxide plate 10
serves as an electrode. A tin oxide film 14 at the other face of the layer serves as the other
30 electrode.

In operation absorption of a photon by the sensitiser dye results in electron injection
into the titanium oxide conduction band. Electron conduction into the conducting glass
contact 14 proceeds through the titanium oxide nanocrystalline particles 4 whilst holes are

5 conducted by the ionic conducting polymer electrode 8 through the pores in the titanium oxide film to the platinum or conducting glass counter electrode 12.

As mentioned previously iodine is mixed in with the ionic conducting polymer electrode. Iodine is a volatile liquid. It would have been expected that the iodine would
10 relatively rapidly evaporate from an unsealed cell rendering it inoperative and an unsealed cell approach unworkable. Surprisingly, it has been found that evaporation from the unsealed cell is slow and that the cell has good stability.

The polyethyleneoxide used as the ionic conducting polymer may have an RMM
15 within the range 10,000 to 50,000 and preferably approximately 20,000. It would have been expected that molecules of such a large size would not have allowed intercalation into the pores between the nanocrystalline titanium oxide particles and therefore only allowed a poor cell efficiency. Surprisingly, it has been found that even such large molecules yield good efficiency which either indicates that intercalation is occurring or that intercalation is
20 unimportant for the efficiency. The polyethyleneoxide has been found to yield a surprisingly high conductivity when formed at a temperature within the range 50°C to 100°C and preferably approximately 70°C and at a pressure within the range of 0.25kg/cm² to 1kg/cm² and preferably approximately 0.5kg/cm².

25 Examples of the invention have achieved efficiencies approximately twice as efficient as the best solid state dye sensitised device reported to date, with excellent device stability. Further refinement is likely to result in further significant improvements in performance. A summary of results to date is attached.

30 The invention is based upon the use of an ionic conducting polymer: polyethyleneoxide/LiI/I₂. The system is solvent free, and stable cell outputs have been observed for in excess of one month without any cell scaling. The efficiencies of the device is further improved by the addition of triaryl amine components as hole conductors.

5

Solid State Electrolytes, Progress 31/3/99

HTM	Solvent	Efficiency 1 sun	Efficiency 0.1 sun	stability
PEG/LiI/I2	Ethanol	0.2%	1%	20% loss over one month
PEG/LiI/I2/TPD	Acetone	0.4%	1.3%	<10% over 1 month
PEG/LiI/I2/PC/EC	None	0.5%	1.4%	Not Done

- Efficiencies measure 2 days after fabrication and after evaporation of solvents. All cells unsealed.
- 10 - Cell Type 2 (+TPD) 2.5% efficient at 0.1 sun, 4 hours after fabrication, but reduced to table values over 2 days, efficiency at 0.1 sun then stabilised for one month (1 sun efficiencies dropped to 0.2%)
- All cells fabricated with transparent TiO₂, no TiO₂ backing layer, nonreflective counter electrode (equivalent liquid electrolyte cell efficiency 5-6%).
- 15 - Best previous published solid state cell data 0.78% at 0.1 sun (Back et al., Nature 1998)

As an alternative to polyethyleneoxide it would be also possible to use molecular hole transporting materials such as TPD and derivatives thereof

20

It will be appreciated that the devices described above may also have bioanalytical applications. Alternatives to metal oxide nanoparticles include semiconductor sensitizer nanoparticles. It is possible that low temperature fabrication techniques would make such material suitable for deposition upon plastics.

5

CLAIMS

1. A photovoltaic device including an ionic conducting material as a solid state electrode.
- 10 2. A photovoltaic device as claimed in claim 1, wherein said ionic conducting material comprises molecular, oligomer or polymeric components.
3. A photovoltaic device as claimed in any one of claims 1 and 2 including a nanocrystalline particle film.
- 15 4. A photovoltaic device as claimed in claim 3, wherein said nanocrystalline particle film is a nanocrystalline metal oxide film.
5. A photovoltaic device as claimed in any one of claims 3 and 4, wherein said ionic
20 conducting material is intercalated with said nanocrystalline particle film.
6. A photovoltaic device as claimed in claim 5, wherein upon absorption of a photon electron conduction proceeds through said nanocrystalline particle film and hole conduction proceeds through said ionic conducting material.
- 25 7. A photovoltaic device as claimed in any one of claims 3 to 6, wherein said nanocrystalline particle film is dye sensitised.
8. A photovoltaic device as claimed in any one of the preceding claims, wherein said
30 ionic conducting material is doped to enhance hole conduction.
9. A photovoltaic device as claimed in any one of the preceding claims, wherein conductivity of said ionic conducting material incorporates electronic hole transporting components.

5

10. A photovoltaic device as claimed in claim 9, wherein tyiaryl amine compounds are added to said ionic conducting material.

11. A photovoltaic device as claimed in any one of the preceding claims, wherein said
10 ionic conducting material is mixed with redox active components.

12. A photovoltaic device as claimed in claim 11, wherein said ionic conducting material is mixed with ionic salts.

13. A photovoltaic device as claimed in claim 12, wherein said ionic salt is lithium
15 iodide.

14. A photovoltaic device as claimed in any one of the preceding claims, wherein said ionic conducting material includes polyethyleneoxide.

20

15. A photovoltaic device as claimed in any one of the preceding claims, wherein said ionic conducting material has an RMM in the range 10,000 to 50,000

16. A photovoltaic device as claimed in any one of the preceding claims, wherein said
25 ionic conducting material has an RMM of substantially 20,000.

17. A photovoltaic device as claimed in claim 12, including a polyethyleneoxide, iodine and iodide salt mix.

18. A photovoltaic device as claimed in claim 3 and any one of claims 4 to 17, wherein
30 said ionic conducting material and said nanocrystalline particle film are disposed on one of a conducting glass substrate or conducting plastic substrate.

5 19. A photovoltaic device as claimed in claim 18, wherein said ionic conducting material is mixed with iodine and said photovoltaic device is unsealed.

20. A photovoltaic device as claimed in claim 3, wherein said nanocrystalline particle film is formed of titanium oxide nanoparticles.

10

21. A photovoltaic device as claimed in claim 3, wherein said nanocrystalline particle film is formed of zinc oxide nanoparticles.

22. A photovoltaic device as claimed in claim 3, wherein said nanocrystalline particle
15 film is formed of nanoparticles of between 5 and 50 nanometers in diameter.

23. A method of making a photovoltaic device, said method comprising the steps of.
forming a porous nanocrystalline particle film; and
forming an ionic conducting material intercalated with said nanocrystalline particle
20 film, said ionic conducting material film serving as a solid state electrode.

24. A method as claimed in claim 21, wherein said porous nanocrystalline particle film is formed by one of.
use of an aqueous sol/gel;
25 thermolysis in a high boiling point solvent;
chemical vapour deposition;
spray pyrolysis; and
spreading and spin coating

30 25. A method as claimed in claim 23, wherein said ionic conducting material is a polyethyleneoxide.

26. A method as claimed in claim 25, wherein said polyethyleneoxide is formed at a temperature in the range 50°C to 100°C

5 27. A method as claimed in claim 25, wherein said polyethyleneoxide is formed at a temperature of substantially 70°C.

28. A method as claimed in claim 25, wherein said polyethyleneoxide is formed at a pressure in the range 0.25kg/cm² to 1kg/cm²,

10

29. A method as claimed in claim 25, wherein said polyethyleneoxide is formed at a pressure of substantially 0.5kg/cm².

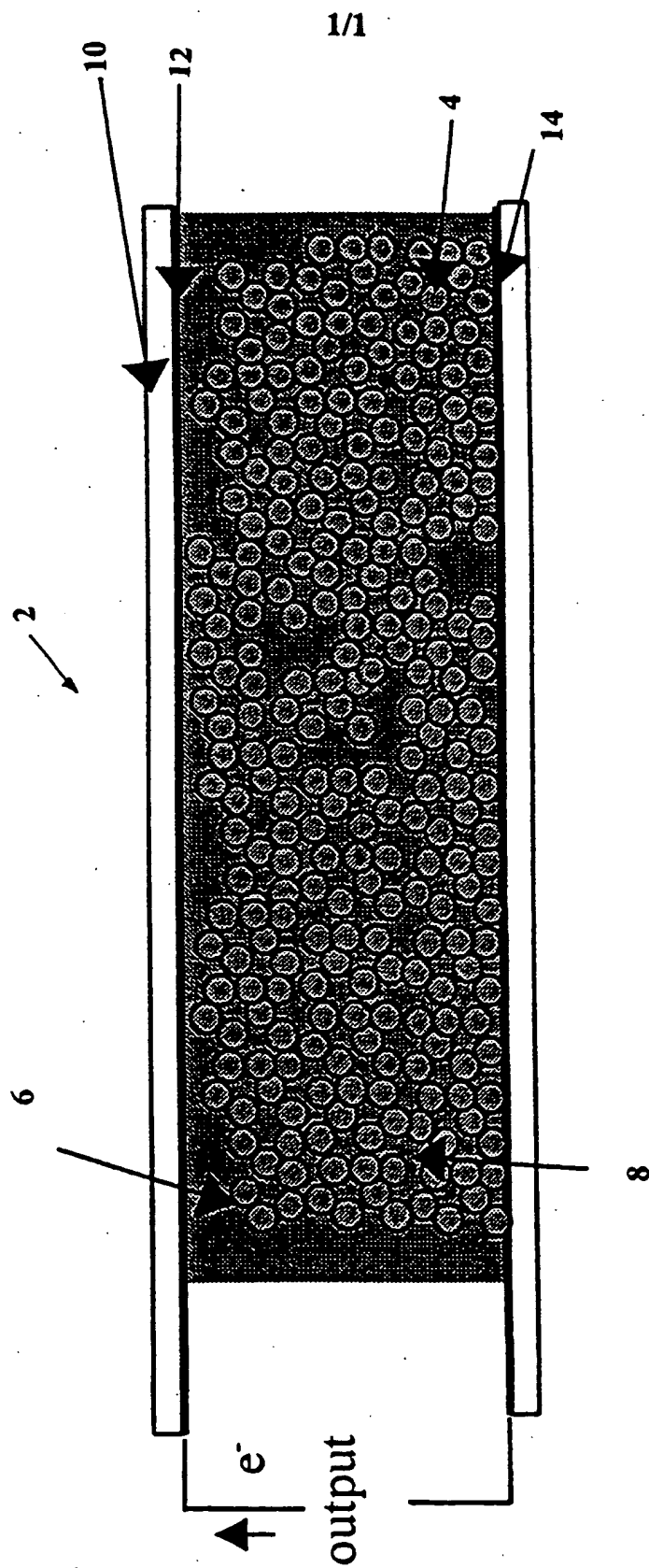


FIGURE 1

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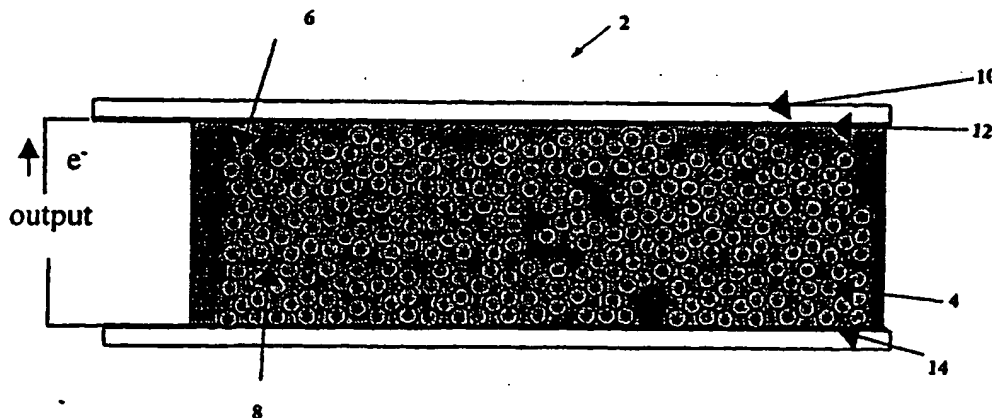
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(54) Title: PHOTOELECTROCHEMICAL CELL WITH A SOLID ELECTROLYTE



(57) Abstract: A photovoltaic device (2) is described comprising a porous film of titanium oxide nanocrystalline particles (4) intercalated with an ionic conducting material (polymer) electrode (8). The ionic conducting polymer electrode (8) may be formed of a mixture of polyethyleneoxide, lithium iodide and iodine. The nanocrystalline titanium oxide particles (4) are dye sensitised. In operation a photon is absorbed injecting an electron into the conduction band of the titanium oxide (4). A hole is passed to the ionic conducting polymer (8). The electron is conducted through the titanium oxide nanocrystalline particles (4) and the hole is conducted through the ionic conducting polymer electrode (8). The cell may be unsealed without rapid evaporation of iodine.

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B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

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Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

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C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 0 720 189 A (ISHIHARA SANGYO KAISHA) 3 July 1996 (1996-07-03) page 3, line 26 - line 56 page 5, line 10 - line 32 ---	1-5, 7, 8, 11-14, 17-20, 22-25
X	L. R. A. K. BANDARA, ET AL.: "Dye Sensitized Photoelectrochemical Solar Cells with PEO Based Solid Polymer Electrolytes" SOLID STATE Ionics: SCIENCE AND TECHNOLOGY, PROC. 6TH ASIAN CONF., - 1998 pages 493-498, XP000933641 Singapore the whole document --- -/--	1-5, 7, 11-14, 17-20, 22-25



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NL - 2280 HV Rijswijk
Tel. (+31-70) 340-2040, Tx. 31 851 epo nl,
Fax: (+31-70) 340-3018

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INTERNATIONAL SEARCH REPORT

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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	MATSUMOTO M ET AL: "A dye sensitized TiO2 photoelectrochemical cell constructed with polymer solid electrolyte" SOLID STATE IONICS, NL, NORTH HOLLAND PUB. COMPANY. AMSTERDAM, vol. 89, no. 3-4, 2 August 1996 (1996-08-02), pages 263-267, XP004069984 ISSN: 0167-2738 the whole document ----	1-5
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P, X	EP 0 973 181 A (FUJI PHOTO FILM CO LTD) 19 January 2000 (2000-01-19) the whole document -----	1-5

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PC 38 00/01475

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
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